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# UTILITY PATENT APPLICATION TRANSMITTAL (Large Entity)

(Only for new nonprovisional applications under 37 CFR 1.53(b))

Docket No.

WLJ.051

Total Pages in this Submission

3

**TO THE ASSISTANT COMMISSIONER FOR PATENTS**

Box Patent Application

Washington, D.C. 20231

Transmitted herewith for filing under 35 U.S.C. 111(a) and 37 C.F.R. 1.53(b) is a new utility patent application for an invention entitled:

**A METHOD OF DEPOSITING LAYERS**

and invented by:

BEEKMAN, Knut

RICH, Paul

WIGGINS, Claire Louise

JC564 U.S. PTO  
09/548014  
04/12/00

If a **CONTINUATION APPLICATION**, check appropriate box and supply the requisite information:

☒ Continuation ☐ Divisional ☐ Continuation-in-part (CIP) of prior application No.: \_\_\_\_\_

Which is a:

☒ Continuation ☐ Divisional ☐ Continuation-in-part (CIP) of prior application No.: \_\_\_\_\_

Which is a:

☐ Continuation ☐ Divisional ☐ Continuation-in-part (CIP) of prior application No.: \_\_\_\_\_

Enclosed are:

**Application Elements**

☒ Filing fee as calculated and transmitted as described below

2. ☒ Specification having 12 pages and including the following:

- a. ☒ Descriptive Title of the Invention
- b. ☐ Cross References to Related Applications (if applicable)
- c. ☐ Statement Regarding Federally-sponsored Research/Development (if applicable)
- d. ☐ Reference to Microfiche Appendix (if applicable)
- e. ☒ Background of the Invention
- f. ☒ Brief Summary of the Invention
- g. ☐ Brief Description of the Drawings (if drawings filed)
- h. ☒ Detailed Description
- i. ☒ Claim(s) as Classified Below
- j. ☒ Abstract of the Disclosure

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## Application Elements (Continued)

3. ☐ Drawing(s) (when necessary as prescribed by 35 USC 113)
- a. ☐ Formal Number of Sheets \_\_\_\_\_
- b. ☐ Informal Number of Sheets \_\_\_\_\_
4. ☒ Oath or Declaration
- a. ☒ Newly executed (original or copy) ☐ Unexecuted
- b. ☐ Copy from a prior application (37 CFR 1.63(d)) (for continuation/divisional application only)
- c. ☒ With Power of Attorney ☐ Without Power of Attorney
- d. ☐ DELETION OF INVENTOR(S)  
Signed statement attached deleting inventor(s) named in the prior application,  
see 37 C.F.R. 1.63(d)(2) and 1.33(b).
5. ☐ Incorporation By Reference (usable if Box 4b is checked)  
The entire disclosure of the prior application, from which a copy of the oath or declaration is supplied under  
Box 4b, is considered as being part of the disclosure of the accompanying application and is hereby  
incorporated by reference therein.
6. ☐ Computer Program in Microfiche (Appendix)
7. ☐ Nucleotide and/or Amino Acid Sequence Submission (if applicable, all must be included)
- a. ☐ Paper Copy
- b. ☐ Computer Readable Copy (identical to computer copy)
- c. ☐ Statement Verifying Identical Paper and Computer Readable Copy

## Accompanying Application Parts

8. ☒ Assignment Papers (cover sheet & document(s))
9. ☐ 37 CFR 3.73(B) Statement (when there is an assignee)
10. ☐ English Translation Document (if applicable)
11. ☐ Information Disclosure Statement/PTO-1449 ☐ Copies of IDS Citations
12. ☒ Preliminary Amendment
13. ☒ Acknowledgment postcard
14. ☐ Certificate of Mailing
- ☐ First Class ☐ Express Mail (Specify Label No.): \_\_\_\_\_

**UTILITY PATENT APPLICATION TRANSMITTAL**  
**(Large Entity)**

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3

**Accompanying Application Parts (Continued)**

15. ☐ Certified Copy of Priority Document(s) (if foreign priority is claimed)

16. ☐ Additional Enclosures (please identify below):

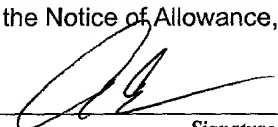
**Fee Calculation and Transmittal**

**CLAIMS AS FILED**

For	#Filed	#Allowed	#Extra	Rate	Fee
Total Claims	16	- 20 =	0	x \$18.00	\$0.00
Indep. Claims	2	- 3 =	0	x \$78.00	\$0.00
Multiple Dependent Claims (check if applicable) <input type="checkbox"/>					\$0.00
BASIC FEE					\$690.00
OTHER FEE (specify purpose) ASSIGNMENT RECORDAL FEE					\$40.00
TOTAL FILING FEE					\$730.00

- ☒ A check in the amount of **\$730.00** to cover the filing fee is enclosed.
- ☒ The Commissioner is hereby authorized to charge and credit Deposit Account No. **50-0238** as described below. A duplicate copy of this sheet is enclosed.
- ☐ Charge the amount of \_\_\_\_\_ as filing fee.
- ☒ Credit any overpayment.
- ☒ Charge any additional filing fees required under 37 C.F.R. 1.16 and 1.17.
- ☐ Charge the issue fee set in 37 C.F.R. 1.18 at the mailing of the Notice of Allowance, pursuant to 37 C.F.R. 1.311(b).

Dated: **APRIL 12, 2000**

  
Signature  
**ADAM C. VOLENTINE**  
**REG. NO. 33,289**

**JONES VOLENTINE, LLP**  
**12200 SUNRISE VALLEY DRIVE, SUITE 150**  
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CC:

**TEL. NO. (703) 715-0870**

IN THE UNITED STATES PATENT AND TRADEMARK OFFICE

In re Patent Application of :  
Knut Beekman et al. : Attn: Applications Branch  
Serial No. [NEW] : Attorney Docket No. WLJ.051  
Filed: April 12, 2000 :  
For: A METHOD OF DEPOSITING LAYERS

**PRELIMINARY AMENDMENT**

Honorable Assistant Commissioner  
of Patents and Trademarks,  
Washington, D.C. 20231

Sir:

Preliminary to the examination of the above-identified application, please enter the following amendments and remarks.

**IN THE CLAIMS**

Please amend the claims as follows:

Claim 4, line 1, change "any on of the preceding claims" to --Claim 1--.

Claim 6, line 1, change "any on of the preceding claims" to --Claim 1--.

Claim 9, line 1, change "any on of the preceding claims" to --Claim 1--.

Claim 13, line 1, delete "or Claim 12".

Claim 14, line 1, delete "or Claim 12".

Claim 15, line 3, change "any one of the preceding claims" to --Claim 1--.

Please add the following new Claim 16:

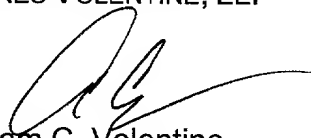
--16. A method of forming an acoustic wave device including depositing a metallic layer in accordance with a method as claimed in Claim 11.--

### REMARKS

By this Preliminary Amendment, the claims have been amended to eliminate multiple dependent claims, and new Claim 16 has been added. Entry of this Preliminary Amendment is respectfully requested.

Respectfully submitted,

JONES VOLENTINE, LLP



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Date: April 12, 200

"A Method of Depositing a Layer"

This invention relates to a method of depositing a layer on an exposed surface of an insulating layer of material.

It has been known for some time that the grain  
5 structure of a deposited layer can be affected by the  
structure of the layer on to which it is deposited. This  
relationship is discussed in terms of aluminium layers  
deposited on titanium in US Patent 5523259 and on titanium  
nitride layers in US Patent 5242860. Perhaps one of the  
10 most complete and recent expositions on the state of the art  
concerning the grain structure of metal conductors on  
barrier layers and how a preferential grain structure is  
achieved is contained in WO 99/10921. However, there is no  
indication in the prior art of the relationship between the  
15 structure of metallic deposited layers and insulating layers  
upon which they lie. Further prior art gives no indication  
of how such an insulating layer may be treated to improve  
the deposited layer structure for these purposes.

In addition to bulk interconnects another technology in  
20 which this is significant is the formation of acoustic wave  
devices wherein the orientation of the piezoelectric layer  
can be significant in the performance of the device.

Thus in a first aspect the invention consists in a  
method of depositing a metallic layer or layers on the  
25 exposed surface of a previously deposited insulating layer  
upon a substrate including treating the exposed surface with

hydrogen or a gaseous source of hydrogen in the presence of a plasma prior to the deposition of the metallic layer or layers.

Surprisingly it has been found that the exposure to  
5 hydrogen changes the structure of at least the exposed surface of the insulating layer in a sense to improve the orientation of a metallic layer and in particular a piezo electric layer subsequently deposited upon the substrate. This may be because hydrogen is implanted in the exposed  
10 surface or because the hydrogen modifies e.g. by etching the exposed surface or a combination of the two.

It is preferred that the extent of the hydrogen treatment is such that the Full Wave Half Maximum (FWHM) of the rocking curve on a preselected crystallographic plane of  
15 a deposited layer is less than  $2.5^\circ$ .

The plasma may be an Inductively Coupled Plasma in which case the substrate may be placed on an RF biased platen, which may be heated. Alternatively the plasma process may be Reactive Ion Etching. In the first case the  
20 process time for the hydrogen treatment may be between 35 and 25 minutes, and in the second case the treatment period may be more than 5 minutes and less than 15 minutes.

Typically the substrate will be a semiconductor such as silicon or the insulating layer will be silicon dioxide.  
25 Where the process is being used in the form of an acoustic wave device, a deposited layer will be preferably required to have a narrow x ray diffraction peak half width on (002) to function as a piezo electric thin film. This deposited

layer is preferably aluminium nitride. It is preferred that the aluminium nitride is deposited at a temperature below 500°C.

As is known in the art the FWHM rocking curve of a diffraction peak is a good indication of degree of orientation. This rocking curve is obtained by rotating a sample in an x-ray beam, which is directed at the surface being inspected. At a particular angle the curve produces a reflectance peak and by rocking the sample about that peak it is possible to determine the angle of rock needed to move the sample from half the maximum intensity on one side of the peak to the corresponding point on the other side of the peak. This angle is referred to as the FWHM measurement and the narrower the angle the better ordered the structure.

In an experiment aluminium nitride was deposited onto an underlayer of aluminium (that forms one electrode) in turn deposited upon a titanium adhesion layer upon an insulting layer of silicon dioxide. The silicon dioxide had been treated in one of three ways and the FWH rocking curve of the aluminium nitride measurement was obtained on (002).

The experimental results were as follows:

Process

<u>characteristic</u>	<u>Standard</u>	<u>Method 1</u>	<u>Method 2</u>
Mode	RF biased	RF biased	RIE
25	ICP	ICP	
Power Inductive coil	350W	350W	-



	Substrate platen	400W	110W	600W
	power			
	Platen temperature	150°C	400°C	400°C
	Process gas	30sccm Ar	50sccm H <sub>2</sub>	300sccm H <sub>2</sub>
5	Process time	32 seconds	30 minutes	10 minutes
	FWHM rocking curve			
	on (002) aluminium			
	nitride peak	5.5	2.4	2.2

All processes here are sputter etching;

10        'ICP' means Inductively Coupled Plasma and includes an  
 RF power supply predominantly inductively coupled to a  
 plasma and an RF power supply connected to the substrate  
 platen.

      'RIE' is used here in its industry standard use;  
 15        meaning that process power is predominantly or exclusively  
 applied to the substrate holder. Any 'reactive' element to  
 the etching is insignificant in the experimental processes  
 reported as silicon dioxide is insignificantly reactive with  
 hydrogen. The predominant etch method is by sputtering and  
 20        due to the low mass of the hydrogen ion the etching is slow.

The standard method is a very short argon etch which is

generally used to clean the surface of a substrate prior to deposition.

The other two methods, which have been developed by the applicants, indicate the benefit of hydrogen pre-treatment, with the FWHM measurement being reduced by over half or, put another way, with an over 100% improvement in orientation.

Although, as has been mentioned above, etching may be a part of what is happening to the surface, it is noted that the etch rate of silicon dioxide using hydrogen is considerably less than argon (~21 Å/min in 'RIE' mode, 6 Å/min in ICP mode compared with 450-650 Å/min in the 'standard' process").

In the light of this, it is considered more likely that hydrogen is implanted into the surface of the substrate improving the grain structure of that surface or it may be that the hydrogen in the surface creates favourable conditions for the grain orientation of the subsequently deposited layer. This process, whilst it enables the formation of bulk SAW devices is commercially viable in

spite of its long process times. However, the applicants have also observed that an 80mm titanium film which is sputtered onto a silicon wafer in a cryogenically pumped vacuum system that has stood idle for a long period of time  
5 (say 10 hours or more) gives a  $\text{Ti}\langle 002 \rangle : \langle 011 \rangle$  XRD ratio that is high ( $>10:1$ ). A vacuum would, in those conditions, have a high hydrogen content due to the well known inability of cryogenic pumps to pump hydrogen well.

Another aspect of the invention is therefore to treat  
10 the first or subsequent metallic layers of a multilayer structure with atomic hydrogen, typically in a plasma. This could most conveniently be done simultaneously with a metallic sputtering process e.g. titanium, titanium nitride, titanium oxide, tungsten, tungsten nitride, tantalum,  
15 tantalum nitride, aluminium, aluminium alloys, copper, aluminium nitride.

The sputtering process requires a plasma to generate the argon ions required for the process from the argon gas supplied to the vacuum process chamber. Hydrogen additions

in controlled small quantities would be ionised by the electric fields present within the vacuum vessel (or could be previously ionised): Thus atomic hydrogen would be incorporated within at least the surface of the sputtered  
5 film causing its crystallographic structure to be preferentially modified.

This structural modification of at least the surface enables a higher proportion of a preferential crystal orientation in a subsequently deposited metal conductor.  
10 Thus a layer of a metallic barrier structure lying between an insulating layer and a conducting layer could be crystallographically modified by the use of this hydrogen treatment thus causing layers further deposited upon it to have a preferential structure. In addition or alternatively  
15 the layers requiring this preferential crystallography could be sputtered themselves in the presence of hydrogen or subsequently treated with atomic hydrogen. The preferential structure is characterised by having increased functional capabilities due to its more regularly ordered

[illegible]

CLAIMS

1. A method of depositing a metallic layer on an exposed surface of previously deposited insulating layer on a substrate including treating the exposed surface with hydrogen or a gaseous source of hydrogen in the presence of a plasma, prior to or during deposition of the metallic layer.
2. A method as claimed in Claim 1 wherein the hydrogen treatment modifies the exposed surface.
3. A method as claimed in Claim 1 wherein hydrogen is implanted in the exposed surface.
4. A method as claimed in any one of the preceding claims wherein the extent of the hydrogen treatment is such that the x-ray diffraction peak half width on a crystallographic plane of a deposited metallic layer is narrowed.
5. A method as claimed in Claim 4 where the metallic layer is aluminium nitride.
6. A method as claimed in any one of the preceding claims wherein the plasma is an Inductively Coupled Plasma.

8. A method as claimed in Claim 7 wherein the platen is heated.

10. A method as claimed in Claim 9 wherein the treatment time is less than 15 minutes.

12. A method as claimed in Claim 11 wherein the metallic layer is deposited by sputtering and molecular hydrogen is added to a metallic sputtering process.

14. A method as claimed in Claim 11 or Claim 12 wherein the

5 15. A method of forming an acoustic wave device including  
depositing a metallic layer in accordance with a method as  
claimed in any one of the preceding claims.



ABSTRACT"A Method of Depositing a Layer"

This invention relates to a method of depositing a layer on an exposed surface of an insulating layer of material. The method includes treating the exposed surface with hydrogen or a gaseous source of hydrogen in the presence of a plasma, prior to or during deposition of a metallic layer.

JONES VOLANTINE, L.L.P. (1/99)

## DECLARATION AND POWER OF ATTORNEY FOR U.S. PATENT APPLICATION

(X) Original ( ) Supplemental ( ) Substitute ( ) PCT ( ) Design

As a below named inventor, I hereby declare that: my residence, post office address and citizenship are as stated below next to my name; that I verily believe that I am the original, first and sole inventor (if only one name is listed below) or an original, first and joint inventor (if plural inventors are named below) of the subject matter which is claimed and for which a patent is sought on the invention entitled:

**TITLE: A METHOD OF DEPOSITING A LAYER**

of which is described and claimed in:

(X) the attached specification, or

( ) the specification in the application Serial No. \_\_\_\_\_ filed \_\_\_\_\_,

and with amendments through \_\_\_\_\_ (if applicable), or

( ) the specification in International Application No. PCT/\_\_\_\_\_, filed \_\_\_\_\_,

and as amended on \_\_\_\_\_ (if applicable).

I hereby state that I have reviewed and understand the content of the above-identified specification, including the claims, as amended by any amendment(s) referred to above.

I acknowledge my duty to disclose information of which I am aware which is material to the examination of this application in accordance with Title 37, Code of Federal Regulations, §1.56(a).

I hereby claim foreign priority benefits under Title 35, United States Code, §119 (and §172 if this application is for a Design) of any foreign application(s) for patent or inventor's certificate listed below and have also identified below any foreign application for patent or inventor's certificate having a filing date before that of the application on which priority is claimed:

COUNTRY	APPLICATION NO.	DATE OF FILING	PRIORITY CLAIMED
GREAT BRITAIN	9908882.5	20 APRIL 1999	YES

I hereby claim the benefit under Title 35, United States Code, §120 of any United States application(s) listed below and, insofar as the subject matter of each of the claims of this application is not disclosed in the prior United States application in the manner provided by the first paragraph of Title 35, United States Code, §112, I acknowledge the duty to disclose material information as defined in Title 37, Code of Federal Regulations, §1.56(a) which occurred between the filing date of the prior application and the national or PCT international filing date of this application:

APPLICATION SERIAL NO.	U.S. FILING DATE	STATUS: PATENTED, PENDING, ABANDONED

And I hereby appoint Raymond C. Jones, Reg. No. 34,631 and Adam C. Valentine, Reg. No. 33,289, members of the firm of JONES VOLLENTINE, L.L.P., jointly and severally, attorneys to prosecute this application and to transact all business in the U.S. Patent and Trademark Office connected therewith.

I hereby authorize the U.S. attorneys named herein to accept and follow instructions from WYNNE-JONES, LAINE & JAMES as to any action to be taken in the U.S. Patent and Trademark Office regarding this application without direct communication between the U.S. attorneys and myself. In the event of a change in the persons from whom instructions may be taken, the U.S. attorneys named herein will be so notified by me.

Kindly direct all correspondence to: **JONES VOLLENTINE, L.L.P.**  
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Residence & Citizenship	CITY	STATE OR COUNTRY	COUNTRY OF CITIZENSHIP
Post Office Address	ADDRESS	CITY	STATE OR COUNTRY ZIP CODE
Full Name of 5th Inventor	FAMILY NAME	FIRST GIVEN NAME	SECOND GIVEN NAME
Residence & Citizenship	CITY	STATE OR COUNTRY	COUNTRY OF CITIZENSHIP
Post Office Address	ADDRESS	CITY	STATE OR COUNTRY ZIP CODE

I further declare that all statements made herein of my own knowledge are true, and that all statements on information and belief are believed to be true; and further that these statements were made with the knowledge that willful false statements and the like so made are punishable by fine or imprisonment, or both, under Section 1001 of Title 18 of the United States Code, and that such willful false statements may jeopardize the validity of the application or any patent issuing thereon.

1st Inventor K. Beckman Date 7<sup>th</sup> April 2000  
Karl Beckman  
 2nd Inventor Paul Rich Date 7 APRIL 2000  
Paul Rich  
 3rd Inventor Claire Louise Wiggins Date 7 April 2000  
Claire Louise Wiggins  
 4th Inventor \_\_\_\_\_ Date \_\_\_\_\_  
 5th Inventor \_\_\_\_\_ Date \_\_\_\_\_

Applicant Reference No.: DBN.101 Any Docket No.: WIJ.051